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L14: Entry 1 of 4 File: USPT Nov 11, 2003

DOCUMENT-IDENTIFIER: US 6645618 B2
TITLE: Aliphatic polyester microfibers, microfibrillated articles and use thereof

Brief Summary Text (4):
Polymeric fibers have been known essentially since the beginnings of commercial polymer development. The production of polymer fibers from polymer films is also well known. Typically, molten polymer is extruded through a die or small orifice in a continuous manner to form a continuous thread. The fiber can be further drawn to create an oriented filament with significant tensile strength. Fibers created by a traditional melt spinning process are generally larger than 15 microns. Smaller fiber sizes are impractical because of the high melt viscosity of the molten polymer. Fibers with a diameter less than 15 microns can be created by a melt blowing process. However, the resins used in this process are low molecular weight and viscosity rendering the resulting fibers very weak. In addition, a post spinning process such as length orientation cannot be used.

Brief Summary Text (5):
Orientation of crystalline polymeric films and fibers has been accomplished in numerous ways, including hot drawing, melt spinning, melt transformation (co)extrusion, solid state coextrusion, gel drawing, solid state rolling, die drawing, solid state drawing, and roll-trusion, among others. Each of these methods has been successful in preparing oriented, high modulus polymer fibers and films. Most solid-state processing methods have been limited to slow production rates, on the order of a few cm/min. Methods involving gel drawing can be fast, but require additional solvent-handling steps. A combination of rolling and drawing solid polymer sheets, particularly polyolefin sheets, has been described in which a polymer billet is deformed biaxially in a two-roll calender then additionally drawn in length (i.e., the machine direction). Methods that relate to other web handling equipment have been used to achieve molecular orientation, including an initial nip or calender step followed by stretching in both the machine direction or transversely to the film length.

Brief Summary Text (6):
The production of macroscopic fibers from films has been established. Liberating fibers from oriented, high-modulus polymer films, particularly from high molecular weight semicrystalline films, has been accomplished in numerous ways, including abrasion, mechanical plucking by rapidly-rotating wire wheels, and impinging water jets to slit the film. Water jets have been used extensively to cut films into flat, wide continuous longitudinal fibers for strapping or reinforcing uses.

Brief Summary Text (7):
Pennings et.al. in "Mechanical properties and hydrolyzability of Poly(L-lactide) Fibers Produced by a Dry-Spinning Method", J. Appl. Polym. Sci., 29, 2829-2842 (1984) described fibers with a fibrillar structure by solution spinning using chloroform in the presence of various additives (camphor, polyurethanes) followed by hot drawing. These fibers showed good mechanical properties and improved degradability in vitro with the fibrillar structure speeding up the hydrolysis of the fiber. The inherent disadvantage of this process is the use of chlorinated solvents in the spinning process.

Brief Summary Text (8):
Microfibers with a diameter of 1 micrometer and a round cross section have also been

produced by electrospinning. The electrospinning technique also suffers from the disadvantage of using a chlorinated solvent and has low production speeds.

Brief Summary Text (10):
U.S. Pat. No. 6,111,060 (Gruber et al.) discloses the use of melt stable polylactides to form nonwoven articles via melt blown and spunbound processes. These fibers have low orientation and have generally low tensile strength. In addition, the fibers have a round cross sectional area comparable to traditional textile fibers.

Brief Summary Text (11):
WO 9824951 discloses the production of multicomponent fibers for nonwovens comprising two different polylactides.

Brief Summary Text (13):
The present invention is directed to aliphatic polyester microfibers having an average effective diameter less than 20 microns, generally from 0.01 microns to 10 microns, and substantially rectangular in cross section, having a transverse aspect ratio (width to thickness) of from 1.5:1 to 20:1, and generally about 3:1 to 9:1. Since the microfibers are substantially rectangular, the effective diameter is a measure of the average value of the width and thickness of the microfibers. The cross-sectional area of the fibers is generally from about 0.05 to 3.0.mu..sup.2, and typically 0.1 to 2.0.mu..sup.2.

Brief Summary Text (14):
The rectangular cross-sectional shape advantageously provides a greater surface area (relative to fibers of the same diameter having round or square cross-section) making the microfibers (and microfibrillated films) especially useful in applications such as filtration and as reinforcing fibers in cast materials. The surface area is generally greater than about 0.25 m.sup.2 /gram, typically about 0.5 to 30 m.sup.2 /g. Further, due to their biodegradability and/or bioabsorbability, the microfibers of the present invention are useful in applications such as geotextiles, as suture materials and as wound dressings for skin surfaces.

Brief Summary Text (17):
Advantageously the process of the invention is capable of high rates of production, is suitable as an industrial process and uses readily available polymers. The microfibers and microfibrillated articles of this invention, having extremely small fiber diameter and both high strength and modulus, are useful as tape backings, strapping materials, films with unique optical properties and high surface area, low density reinforcements for thermosets, impact modifiers or crack propagation prevention in matrices such as concrete, and as fibrillar forms (dental floss or nonwovens, for example). The microfibers and microfibrillated articles may be used in applications where biodegradability and or bio-absorbability are desirable. Such applications include, bandages, and wound dressings, packaging materials such as bags, tape or cartons, personal hygiene products, and geotextiles, such as those used for stabilization, protection or drainage of soils.

Brief Summary Text (18):
The process of the invention produces a fiber having a high degree of uniaxial orientation resulting in high strength, modulus, and toughness compared to prior art processes for producing microfibers. Furthermore, the process does include the use of solvents that are costly and possibly harmful. The fibers also have a unique cross sectional aspect ratio .gtoreq.1.5 and an effective diameter of less than ten micrometers, generally less than 5 micrometers.

Detailed Description Text (7):
Examples of aliphatic polyesters include those homo-and copolymers derived from (a) one or more of the following diacids (or derivative thereof): succinic acid, adipic acid, 1,12 dicarboxydodecane, fumaric acid, and maleic acid and (b) one of more of the following diols: ethylene glycol, polyethylene glycol, 1,2-propane diol, 1,3-propanediol, 1,2-propanediol, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 1,6-hexanediol, diethylene glycol, and polypropylene glycol, and (c) optionally a small amount, i.e. 0.5-7.0 mole % of a polyol with a functionality greater than two such as glycerol, neopentyl glycol, and pentaerythritol.

Detailed Description Text (22):
Generally, the greater the microvoid (or void) content, the greater the ease of microfibrillation by the process of this invention. Microfibrillation can be defined as the process of breaking a film down into its microfibrillar components where the microfibers are generally less than 10 microns in average fiber diameter. Preferably, when preparing an article having at least one microfibrillated surface, at least one major surface of the polymer film should have a microvoid content in excess of 5%, preferably in excess of 10%, as measured by density; i.e., the ratio of the density of the microvoided film with that of the starting film. Microvoided films useful in the present invention may be distinguished from other voided films or articles, such as microporous films or foamed articles in that the microvoids are generally non-cellular, relatively planar and have major axes in the machine direction (direction of orientation) of the film. The microvoids do not generally interconnect, but adjacent microvoids may intersect.

Detailed Description Text (45):
Generally, greater void content enhances the subsequent microfibrillation, and subsequently, using the process of this invention, for uniaxially oriented films, the greater the yield of fibers. Preferably, when preparing an article having at least one microfibrillated surface, the polymer film should have a void content in excess of 5%, more preferably in excess of 10%, as measured by density; i.e., the change in density devided by the initial density; (.delta..sub.initial -.delta..sub.final)/.delta..sub.initial. Unexpectedly, it has been found that voids may be imparted to the two component (aliphatic polyester and void initiating) polymer films under condition far less severe than those necessary to impart microvoids to microvoided films previously described. It is believed that the immiscible blend, with limited solubility of the two phases and a free energy of mixing greater than zero, facilitates the formation of the voids necessary for subsequent microfibrillation. The voiding is further aided by the lower orientation temperature utilized in the first orientation stage.

Detailed Description Text (62):
The voided or microvoided aliphatic polyester film is then microfibrillated by imparting sufficient fluid energy to the surface to release the microfibers from the polymer matrix. In a microfibrillation process, relatively greater amounts of energy are imparted to the film surface to release microfibers, relative to that of a conventional mechanical fibrillation process. Microfibers are several orders of magnitude smaller in diameter than the fibers obtained by mechanical means (such as with a porcupine roller) ranging in size from less than 0.01 microns to 20 microns. The microfibers obtained from uniaxially oriented films are rectangular in cross section, having a cross sectional aspect ratio (transverse width to thickness) ranging from of about 1.5:1 to about 30:1. Further, the sides of the rectangular shaped microfibers (prepared from uniaxially oriented films) are not smooth, but have a scalloped appearance in cross section. Scanning electron microscopy reveals that the microfibers of the present invention are bundles of individual or unitary microfibrils, which in aggregate form the rectangular or ribbon-shaped microfibers. Thus the surface area exceeds that which may be expected from rectangular shaped microfibers, and such surface enhances bonding in matrices such as concrete and thermoset plastics, as well as provide greater surface area for enhanced biodegradability, where desired.

Detailed Description Text (63):
Optionally, prior to microfibrillation, the film may be subjected to a macrofibrillation step by conventional mechanical means to produce macroscopic fibers from the highly oriented film. The conventional means of mechanical fibrillation uses a rotating drum or roller having cutting elements such as needles or teeth in contact with the moving film. The teeth may fully or partially penetrate the surface of the film to impart a macrofibrillated surface thereto. Other similar macrofibrillating treatments are known and include such mechanical actions as twisting, brushing (as with a porcupine roller), rubbing, for example with leather pads, and flexing. The fibers obtained by such conventional macrofibrillation processes are macroscopic in size, generally several hundreds of microns in cross section. Such macroscopic fibers are useful in a myriad of products such as particulate filters, as oil absorbing media, and as electrets.

Detailed Description Text (68):
The jet(s) may be configured such that all or part of the film surface is microfibrillated. Alternatively, the jets may be configured so that only selected areas of the film are microfibrillated. Certain areas of the film may also be masked, using conventional masking agents to leave selected areas free from microfibrillation. Likewise the process may be conducted so that the microfibrillated surface penetrates only partially, or fully through the thickness of the starting film. If it is desired that the microfibrillated surface extend through the thickness of the film, conditions may be selected so that the integrity of the article is maintained and the film is not severed into individual yams or fibers. A screen or mesh may be used to impart a pattern to the surface of the microfibrillated article.

Detailed Description Text (82):
Optionally the microfibers may be harvested from the surface of the film by mechanical means such as with a porcupine roll, scraping and the like. Harvested microfibers generally retain their bulkiness (loft) due to the high modulus of the individual microfibers and, as such, are useful in many thermal insulation applications such as clothing. If necessary, loft may be improved by conventional means, such as those used to enhance the loft of blown microfibers, for example by the addition of staple fibers.

Detailed Description Text (87):
The present invention provides microfibers with a very small effective average diameter (average width and thickness), generally less than 10 .mu.m) from aliphatic polyester materials. The small diameter of the microfibers provides advantages in many applications where efficiency or performance is improved by small fiber diameter. For example, the surface area of the microfibers (or the microfibrillated film) is inversely proportional to fiber diameter allowing for the preparation of more efficient filters. The high surface area also enhances the performance when used as adsorbents, such as in oil-absorbent mats or batts used in the clean up of oil spills and slicks. Such performance advantages are enhanced when using charged microfibers, fibers and microfibrillated articles of the present invention.

Detailed Description Text (89):
The wipe (or wiping article) may also be prepared from the microfibers harvested from the microfibrillated article. Such fibers may be used for example, in a non-woven construction using techniques known to the art. Such a non-woven construction may further include stable fibers.

Detailed Description Text (92):
When used as a filtration media, the microfibrillated article may be used in complex shapes, such as pleats. Pleated structures may be prepared by standard pleating methods and equipment. The filtration media may be used alone or may be laminated to further functional layers by adhesives, heat bonding, ultrasonics and the like. The further functional layers can be prefilter layers for large diameter particles, support layers such as scrims, spunbond, spunlace, melt blown, air-laid nonwoven, wet laid nonwoven, or glass fiber webs, netting such as Delnet, metal mesh or the like; absorbant filter media, or protective cover layers. Multiple layers of the filter media may be laminated together to provide improved performance.

Other Reference Publication (2):
J. W. Leenslag et al., "Resorbable Materials of Poly(L-lactide). V. Influence of Secondary Structure on the Mechanical Properties and Hydrolyzability of Poly(L-lactide) Fibers Produced by a Dry-Spinning Method", Journal of Applied Polymer Science, vol. 29, pp. 2829-2842, (1984).

Other Reference Publication (8):
H. Steuer, "Biohybride Nerve Guide for Regeneration: Degradable Polylactide Fibers Coated With Rat Schwann Cells", Neuroscience Letters 277, pp. 165-168, (1999).

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